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AN INVESTIGATION OF THE PROPERTIES OF HIGH VALUED RESISTORS AND METHODS OF REDUCING SURFACE LEAKAGE

by

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AN INVESTIGATION OF THE PROPERTIES OF HIGH VALUED RESISTORS

AND METHODS OF REDUCING SURFACE LEAKAGE

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ABSTRACT

Resistors are needed which will remain constant in value over a period of months with varying conditions of temperature and humidity. Victoreen resistors of the order of 10^8 to 10^{11} ohms were tested for these characteristics as well as for changes due to continuous current and for polarization. A means of eliminating changes due to humidity was tested on the resistors and on various insulators. This report gives results for the first three months of an investigation begun September 20, 1943.

* * * * *

Since the successful operation of DC amplifiers often depends upon the constancy of value of the high resistors used in the circuit, it was decided to make an investigation of the properties of available resistors. Preliminary experiments showed the S. S. White resistors to have a temperature coefficient of one or two per cent per degree Centigrade. (See Table 2, also Report CT-693.) These resistors also failed to remain constant in value over a period of several months. The IRC metallized resistors tested showed polarization effects.

The resistors obtained from the Victoreen Instrument Company seemed to be the best available and were chosen for further investigation, to be carried out over a period of several months. Those used ranged in value from 10^8 to 10^{11} ohms. The Victoreen resistor is enclosed in a partially evacuated glass tube. At one end a bead of insulating material is cemented to the glass for the purpose of decreasing surface conduction. (See Figure 1.)

Figure 1. Victoreen Resistor. 1. Glass case, 2. cement, 3. bead of insulating material.

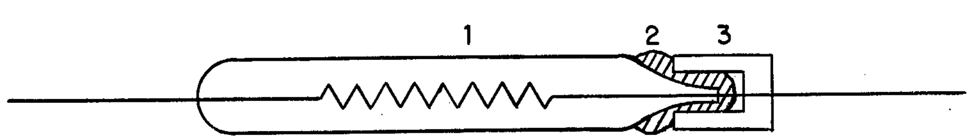

Figure 1

Tests were made for the purpose of determining the following:

1. Constancy of value over a period of months.
2. Effect of aging by prolonged heating at 60°C.
3. Temperature coefficients.
4. Changes due to humidity.
5. Effect of varnish in reducing any change due to humidity.
6. Effect of continuous current at higher voltages over a period of months; polarization effects.

A Lindemann electrometer was used for making resistance measurements. The circuit is shown in Figure 2.

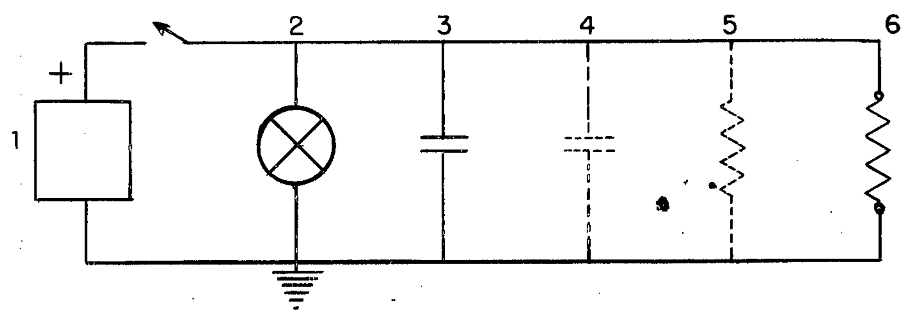

Figure 2. Circuit for measuring resistance. 1. Leeds and Northrup potentiometer, 2. Lindemann electrometer, 3. condenser, 4. capacity of the system, 5. resistance of the system, 6. unknown resistance.

A small voltage is applied to the system, charging the condenser. When the switch is opened the condenser discharges through the resistor to be measured according to the following relationship:

$v = v_o e^{-t/RC}$ where v_o is the voltage when time, t, = 0. C = total capacity in farads, R = resistance in ohms.

Then $\frac{v_o}{v} = e^{t/RC}$

$$\ln \frac{v_o}{v} = \frac{t}{RC}$$

$$R = \frac{t}{C \ln \frac{v_0}{v}}$$

An approximate value for R may be obtained from the following: taking the derivative of the original equation:

$$\frac{dv}{dt} = \frac{-V_0}{RC} e^{-t/RC}$$

dividing by the first equation:

$$\frac{\frac{dv}{dt}}{v} = -\frac{1}{RC}$$

then $R = vdt/Cdv$ or $v\,\Delta t/C\Delta v$, where Δt is the time required for the voltage to drop Δv, and v is the average voltage for the interval.

For the increment of voltage used, this formula gives a value for R accurate to within 0.4 per cent.

A value of one volt was chosen for v_0 since this is approximately the voltage impressed across these resistors when used in the input stage of the DC amplifiers. The capacity, C, which was the total of the capacity of the system plus that of a condenser, was chosen to give a convenient rate of discharge. For resistors of 10^{12} ohms and above and for insulators, the capacity of the electrometer system alone was sufficient. This capacity was calculated to be about 15 $\mu\mu f$ by extrapolation from a curve where the time of discharge for 0.2 volt was plotted against the capacity added to that of the system. (See Figure 3.)

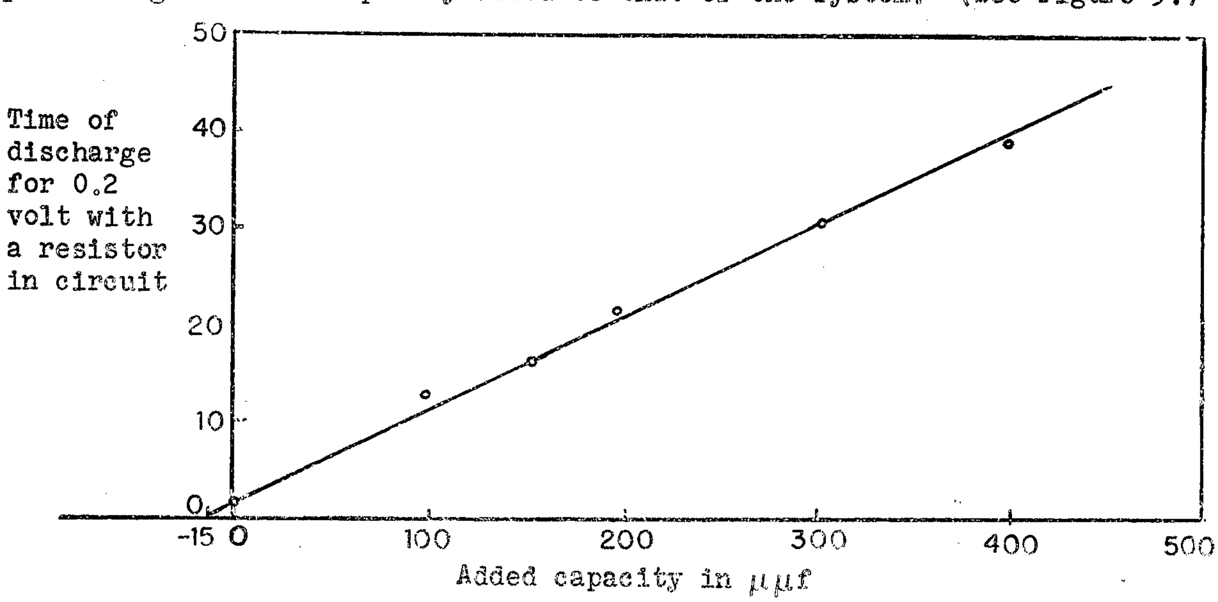

Figure 3. Curve for obtaining capacity of electrometer system.

For added capacity, a Leeds and Northrup air condenser with quartz insulation was used for the values from 50 to 1300 $\mu\mu f$. A mica condenser measuring 0.01025 μf and a Leeds and Northrup 0.025 to 1.0 μf condenser were used for the higher values. It was necessary to keep the quartz insulation of the air condenser dry to prevent surface leakage. This was done by means of phosphorous pentoxide (P_2O_5).

Since the resistance of the electrometer system is in parallel with any resistance measured, it should be kept as high as possible. Polystyrene was used for insulating the leads to the electrometer. The circuit was enclosed in a metal box where the humidity was kept low by means of drying agents. The resistance of the system varied from a little less than 10^{14} ohms when the tests were begun in September to well above 10^{14} ohms in December. This change, however, does not appreciably affect measurements of the order of 10^8 to 10^{11} ohms.

It was necessary to have electrostatic shielding for all leads to the electrometer. This was provided by the metal box mentioned above.

Results obtained during the first three months of the investigation follow. These results are for Victoreen resistors with the exception of a table of temperature coefficients for S. S. White resistors included for comparison.

1. Constancy of value

Forty Victoreen resistors, ten each of the order of 10^8, 10^9, 10^{10}, and 10^{11} ohms, were measured over a period of three months. At the end of this time, twenty-two of them were found to measure within one or two per cent of their original values. Of the rest, all were within the limits of reasonable error, that is, four or five per cent, except two. One 10^8 ohm resistor had increased by about 40 per cent and one 10^{11} ohm resistor had increased by about 100 per cent.

2. Aging by heating at 60°C

Twelve Victoreen resistors, three of each group mentioned above, were kept in an oven at 60°C for three months. This temperature is well above that at which they are used in the DC amplifiers. If there is any effect of aging under actual working conditions, it should become apparent in this test. A voltage of 1.5 volts was kept impressed across two of the 10^9 ohm resistors for the period of test. When the twelve resistors were remeasured at room temperature, eight showed decreases in value of five per cent or less, three had dropped seven, twelve, and twenty per cent, respectively, while one had increased by twenty-four per cent. The two across which 1.5 volts were impressed decreased by one and five per cent, respectively. All significant variations were negative except one.

3. Temperature coefficient

Twelve Victoreen resistors, three of each group, were measured at 23°C, again at 40°C and at 60°C. One of each group was measured at 90°C. Resistances and temperature coefficients are given in Table 1.

At 40°C, no perceptible change from values at room temperature was noted. Of the slight variations, some were positive, some were negative, and all were well within errors of measurement. Coefficients calculated range from 0 to 0.14 per cent per degree Centrigrade with the exception of one positive coefficient of 0.34 per cent per degree Centigrade. For the interval from 40° to 60°C, six resistors showed a mean coefficient of 0 to about -0.2 per cent and six ranged from -0.4 to -0.8 per cent per degree Centigrade. The four heated to 90°C had coefficients varying from -0.15 to -0.71 per cent for the interval 60° to 90°C. (See Figures 4 and 5 for resistor No. 37, which was typical.)

Victoreen resistors seem to be far superior to S. S. White resistors with respect to temperature coefficient in the range of temperature at which they probably will be used. For thirty-six S. S. White resistors, the average temperature coefficient for the interval 23° to 40°C was -1.4 per cent per degree Centigrade. Temperature coefficients of a few representative S. S. Whites are given for comparison. (See Table 2.)

4. Humidity effects

Six Victoreen resistors, 10^8 to 10^{11} ohms, were placed in a can where the humidity could be kept at approximately 95 per cent by an aqueous solution of sodium phosphate (Na_2HPO_4). Holes were drilled in the can so that measurements could be made while the resistors were in the moist atmosphere. Although no change was observed the first day, after a week all the resistors had dropped in value to the order of 10^8 or 10^9 ohms. During the next four weeks, it was found that all decreased gradually to values of 10^7 ohms regardless of original value.

Since humidity effects were so pronounced at 95 per cent, it was decided to see what effect 75 per cent humidity would have. An aqueous solution of sodium acetate ($NaC_2H_3O_2$) was used. Of eleven 10^8 to 10^{11} ohm resistors tested, six dropped by factors of 10 to 100 in two weeks, the 10^{10} and 10^{11} ohm resistors decreasing to 10^9 ohm values. The decrease in these resistors was also gradual.

Three resistors on which the manufacturer used a cement called "aryline", instead of the DeKhotinsky cement otherwise used for cementing the insulating bead to the glass, were tested at 100 per cent humidity. All were of the order of 10^{11} ohms. One decreased to 10^9 ohms and the others to 10^{10} ohms in a week.

Table 1. Temperature coefficients of Victoreen resistors. Mean coefficients in per cent per degree Centrigrade based on lower resistance in each temperature range.

No.	Resistance T_1 23°C	Resistance T_2 40°C	Mean Coef 23° - 40°C	T_3	R_3	Mean Coef 40° - T_3	T_4	R_4	Mean Coef T_3 - T_4
3	$1.35x10^8$	$1.35x10^8$	0	62°	$1.32x10^8$	0.10			
4	$7.90x10^7$	$8.35x10^7$	+0.34	61°	$3.35x10^7$	0.			
12	$4.10x10^8$	$4.2x10^8$	+0.14	61°	$3.85x10^8$	-0.40	93°	$3.3x10^8$	-0.41
19	$1.85x10^9$	$1.85x10^9$	0.	60°	$1.77x10^9$	-0.22			
28	$2.22x10^9$	$2.24x10^9$	+ .05	60°	$2.14x10^9$	-0.22			
37	$4.84x10^9$	$4.84x10^9$	0.	65°	$4.60x10^9$	-0.25	90°-95°	$3.7x10^9$	-0.71
45	$1.47x10^{10}$	$1.44x10^{10}$	-0.12	70°	$1.33x10^{10}$	-0.16			
60	$6.50x10^{10}$	$6.60x10^{10}$	+ 0.09	62°	$6.0 x10^{10}$	-0.41			
61	$7.50x10^{10}$	$7.60x10^{10}$	+ 0.08	70°	$6.7 x10^{10}$	-0.70	90°	$6.5x10^{10}$	-0.15
71	$9.2 x10^{10}$	$9.4 x10^{10}$	+ 0.13	62°	$8.5 x10^{10}$	-0.44			
72	$1.0 x10^{11}$	$1.0 x10^{11}$	0.	62°	$8.25x10^{10}$	-0.80			
73	$1.77x10^{11}$	$1.80x10^{11}$	-0.10	63°	$1.63x10^{11}$	-0.41	85°	$1.33x10^{11}$	-0.61

Table 2. Temperature coefficients of S. S. White resistors. Mean coefficients in per cent per degree Centigrade of ten representative resistors chosen from a group of thirty-six tested.

No. of Resistor	T_1	R_1	T_2	R_2	Mean Coefficient T_1-T_2
2	26°C	$1.03x10^{10}$	45°C	$8.8x10^9$	-0.77
3	26°	$1.09x10^9$	45°	$9.4x10^8$	-0.71
14	26.5°	$9.9 x10^9$	42°	$7.5x10^9$	-1.56
21	26.5°	$3.15x10^{10}$	43°	$2.19x10^{10}$	-1.85
23	26.5°	$3.28x10^{10}$	40.5°	$2.35x10^{10}$	-2.02
28	22°	$1.16x10^{12}$	39°	$6.97x10^{11}$	-2.33
29	22°	$1.01x10^{12}$	44°	$5.08x10^{11}$	-2.26
30	22°	$1.27x10^{12}$	44°	$4.61x10^{11}$	-2.87
31	22°	$1.44x10^{11}$	40.5°	$1.02x10^{11}$	-1.58
33	22°	$1.35x10^{11}$	43°	$1.24x10^{11}$	-0.39

All of these resistors were found to recover their original values within a few minutes after being removed from the moist atmosphere. When replaced, they fell quickly, some in a day, some in a few days, to the low values reached before, instead of following the original pattern of slow decrease. These results may be explained by postulating the rapid formation of a moisture film on the glass followed by slower formation of a film of moisture between the glass and cement, or perhaps over the surface of the cement and between the cement and insulating bead cemented onto one end of the resistor by the manufacturer. (See Figure 1.) When the resistor is removed from the can, we may suppose that the rapid drying of the glass breaks this low resistance path, enabling the resistor to regain its original value. If the slowly formed film between glass and cement or between cement and bead remains, then the resistance may decrease again quite rapidly as soon as the free glass surface acquires a film of moisture.

Of forty resistors tested at 75 to 100 per cent humidity, thirty decreased in resistance by factors of 10 to 1000, the higher resistors by the greater factors. Decrease was noticed in all twelve of the 10^{11} ohm resistors tested, in fourteen of the twenty-two 10^{11} ohm resistors, in three of five 10^9 resistors, and in the only 10^8 ohm resistor tested. The fact that one group of 10^{10} ohm resistors remained constant may indicate that when a good grade of well-cleaned polystyrene is used for the bead and when it is cemented to the glass in such a way that the formation of an interface of moisture is prevented, good results can be obtained.

5. Effect of varnish in reducing surface leakage

A water repellent varnish, No. 9978, manufactured by General Electric Company, was used on the glass surface of the Victoreen resistors, on glass microtubes made by the Victoreen Company, on glass insulators, on selector switches made of steatite, and on porcelain tube bases. All surfaces treated were first cleaned with carbon tetrachloride and baked at high temperatures for the purpose of driving out any adsorbed moisture, the temperature used depending upon the material.

In the first test of the varnish on resistors, the insulating beads were left in place, since it was not known until later that they could be dispensed with. Since the resistors could not be heated above 70°C without warping the bead the sixteen resistors tested were baked at 60°C overnight and heated to 70°C for an hour. The glass surfaces of thirteen of these were varnished with G. E. No. 9978 varnish used with catalyst. The other three were dipped in melted ceresin, coating the bead as well as the glass. Of these, the varnished resistors retained their original values in an atmosphere of 95 to 100 per cent humidity over a period of two weeks. (It was subsequently found that they did not change during several months.) Two of the waxed resistors

showed some decrease in the two-weeks period. This is not to be considered a test of ceresin coatings. These resistors were heated to only 70°C, which probably is not sufficient to prevent an interface of moisture.

From three resistors, beads and cement were removed so they could be heated to 120°C. Higher temperatures sometimes affect the value of the resistors. These resistors were baked for three hours and varnished as above. When subjected to the 95 to 100 per cent humidity test for two weeks, they did not show any appreciable change in value. Similar tests were made on glass microtubes manufactured by the Victoreen Company. Beads and cement were removed from the bases of five of these tubes. They were baked at 110° to 120°C, varnished, and tested as above in a 95 to 100 percent humid atmosphere for two months. Resistance between pins about 2 mm apart remained above 10^{13} ohms. Five untreated tubes fell to values of the order of 10^9 and 10^{10} ohms in the same test. Glass resistors baked at 120°C and varnished showed no leakage that could be detected when subjected to the 100 per cent humidity test for two days.

It was more difficult to get good results with the selector switches made of steatite, which is more porous. Baking at 120°C for three hours before varnishing did not prevent some leakage with exposure to 100 per cent humidity. Some of them fell from more than 10^{14} ohms to 10^{12} ohms in a day. Good results were obtained, however, by baking them at 165°C for seven hours before varnishing. These switches were rebaked after varnishing, at 140°C for sixteen hours, although less time probably would have been sufficient. Seven switches and two porcelain tube bases given the same treatment show no observable leakage after three weeks of the moisture test. One selector switch was soaked in water for eighteen hours. After it was allowed to dry for a half hour, no leakage could be observed. Subsequent tests of this switch at 100 per cent humidity for three weeks failed to show leakage.

6. Effect of higher voltages and polarization effects

Five resistors of the order of 10^8 to 10^{11} ohms were kept with 180 volts impressed across them for three months. They were disconnected occasionally and measured by the original method described, using one volt. Three, of values 10^9, 10^{10}, 10^{11} ohms, remained constant in value. A 10^8 ohm resistor increased gradually to 133 per cent of its original value and a 10^{10} ohm resistor increased to 220 per cent by the end of the month. They then remained comparatively constant.

To test for polarization, each resistor was measured as mentioned in the preceding paragraph, but this time with terminals reversed. Measurements were made as soon as possible after disconnecting the high voltage. No appreciable change was noted.

CONCLUSION

Victoreen resistors may be expected to remain constant in value over a period of several months, at least, and over the range of temperature at which they are likely to be used.

Any changes in value due to humidity in resistors of 10^{12} ohms or less apparently can be eliminated by cleaning, baking for several hours at 120°C, and varnishing with G. E. varnish No. 9978. Further tests of this varnish on glass resistors and on glass microtubes where the leakage path was about 2 mm. in length gave good results. It was successful on steatite and porcelain, where the leakage path was several mm. in length, when higher temperatures and longer periods of baking time were used.

Sustained voltages of the order of 180 volts may affect the Victoreen resistors. When required to carry continuous current, they may change in value but they probably do not become polarized. Since only five resistors were tested for this effect, general conclusions can not be made.

Victoreen resistors may be considered very satisfactory for use in DC amplifiers, particularly when properly coated with G. E. varnish No. 9978.

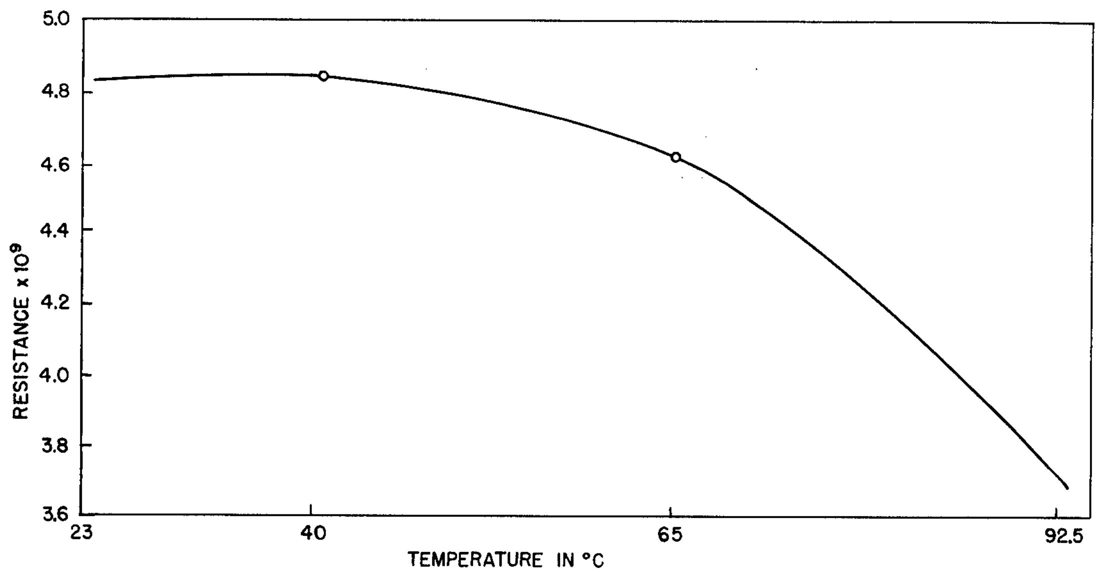

Figure 4. Resistance vs. temperature for resistor No. 37.

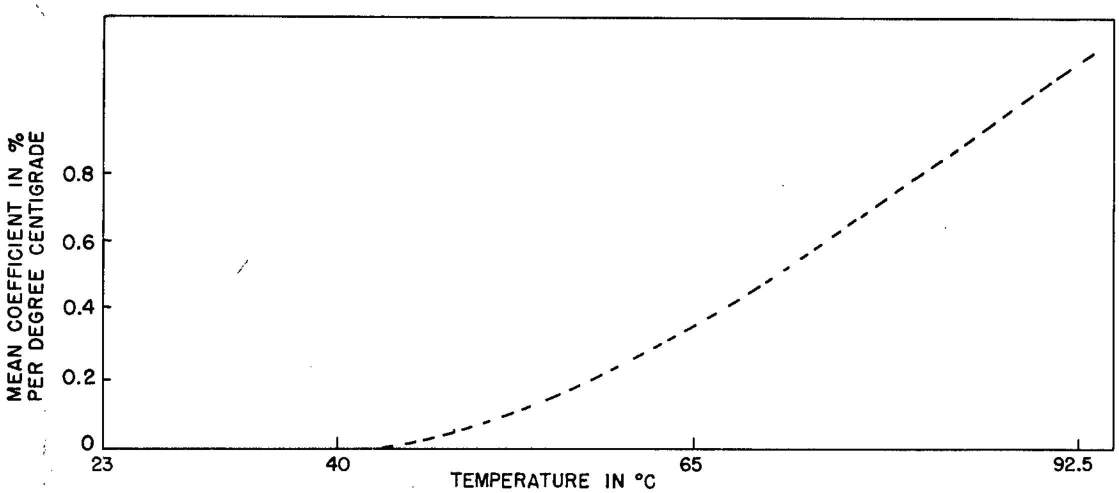

Figure 5. Mean temperature coefficient vs. temperature intervals for resistor No. 37.

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